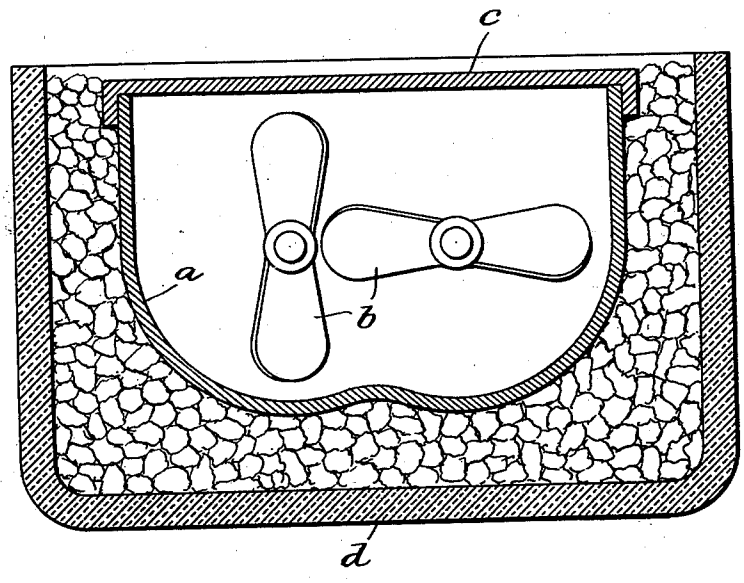
No. 673,377. Patented May 7, 1901.

J. B. BERNADOU.

COLLOID EXPLOSIVE AND PROCESS OF MAKING SAME.

(Application filed Jan. 4, 1901.)

(No Model.)

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# UNITED STATES PATENT OFFICE.

JOHN B. BERNADOU, OF THE UNITED STATES NAVY.

## COLLOID EXPLOSIVE AND PROCESS OF MAKING SAME.

SPECIFICATION forming part of Letters Patent No. 673,377, dated May 7, 1901.

Application filed January 4, 1901. Serial No. 42,064. (No specimens.)

*To all whom it may concern:*

Be it known that I, JOHN B. BERNADOU, lieutenant United States Navy, have invented certain new and useful Improvements in Colloids and Processes of Making the Same, of which the following is a specification.

My invention relates to an improved colloid which may be used after drying as a gunpowder or as an explosive cementing agent or binder in connection with other materials used in the manufacture of smokeless powders, and to process for making the same.

I have discovered that if insoluble nitrocellulose, which I define as nitrocellulose insoluble at ordinary atmospheric temperatures in a mixture of two parts, by weight, of ethylic ether and one part, by weight, of ethyl-alcohol and possessing a content of oxygen above that corresponding to the formula $C_{12}H_{15}O_5(NO_3)_5$, be immersed in ethyl-ether and be exposed to a very low temperature, below that of freezing ethyl-alcohol ninety-five per cent., by volume, absolute, it will go into solution or form a jelly with the ether, resulting in the formation of a colloid. Once in solution or jelly the insoluble nitrocellulose will not again revert to its original form, but constitutes a body which may be employed when dried as a powder or as a cementing agent or binder in the manufacture of powders containing other ingredients.

The property of dissolving in ether in the presence of cold is common to all forms of nitrocellulose. I have hitherto called attention to the solubility of ether-alcohol-soluble nitrocellulose in ether alone in the presence of a moderate degree of cold, (see Letters Patent of the United States issued to me June 26, 1900, No. 652,455,) but now call attention to the solubility in ether alone of the highly-oxygenated or so-called "insoluble" varieties of nitrocellulose upon the application of more intense cold.

At present it is the general practice in the manufacture of colloid smokeless powders to mix the nitrocellulose with a colloiding agent, as acetone or ether-alcohol, to thoroughly mix them in a mechanical mixer and to form the resultant pasty mass into the desired shapes, as rods, strips, or grains, which are then exposed to the air or to a moderate heat until the excess of solvent is expelled. The drying heat must be regular and the drying conducted with care and uniformity. If the powder be dried too quickly, as by exposure to too great a heat, the surface of the grains will be formed into a pellicle, while part of the interior content of the solvent will vaporize, causing the grain to swell and split.

If the powder be not dried uniformly, the damper grains will ignite more slowly than the drier ones, and hence irregularity in ignition and ballistic performance will result. If the powder be left in an undried state, the traces of water, alcohol, and ether remaining in it operate to render it, in comparison with other lots of the same powder properly dried, slow in ignition.

Experience has shown that the complete and uniform drying of the colloid must be effected if the resultant mass is to prove an effective powder. The more effectually that the residual solvent—that remaining after the pasty colloid has been formed into the final shape—can be driven off the more effective will be the powder, the greater its ballistic power, and the less the chance of hang-fires.

As heretofore employed in the manufacture of smokeless powders, insoluble nitrocellulose has been colloided in acetone or in certain other solvents in which it dissolves with ease at ordinary atmospheric temperatures; but such colloids, being brittle, are unsuitable for use as smokeless powders, as they give rise to irregular bore-pressures on discharge of the gun.

I have found by experiment that in the presence of a very intense degree of cold, below that required to freeze ethyl-alcohol ninety-five per cent. absolute by volume, insoluble nitrocellulose can be made to dissolve in ethyl-ether alone. This action of insoluble nitrocellulose may easily be shown by placing a few tenths of a grain of some form of insoluble nitrocellulose—*e. g.*, unpulped gun-cotton—in a test tube, pouring over the same ten or fifteen cubic centimeters of ethylic ether, closely corking the tube, and immersing it in a bath of liquid air. By immersion in a bath of liquid air I mean immersion of the closed vessel containing the insoluble nitrocellulose and ether in a bath of liquid air and exposure of the said vessel to the action of

the liquid air until its contents are reduced to a temperature below that of the freezing-point of ethyl-alcohol ninety-five per cent. absolute. Upon allowing the tube to remain in the liquid-air bath a sufficient length of time, removing it, and allowing its temperature to rise by contact with the atmosphere it will be found that the cotton has lost its fibrous structure and has become converted into a gelatinous body or, if the ether be in large excess, into a syrupy fluid.

The ease with which the insoluble nitrocellulose can be colloided in ether in the presence of cold is materially increased by mechanical agitation—that is, by kneading or incorporating the ether and insoluble nitrocellulose together in a closed vessel, whereby the colloid can be developed by the expenditure of a minimum amount of solvent.

The process of forming the colloid powder therefore consists in subjecting the ether and insoluble nitrocellulose to mechanical action, agitation, or kneading in some approved form of mechanical mixer—such as the Werner and Pfleiderer, with cover, which is herewith illustrated—which is surrounded in whole or in part by a cooling-jacket.

In the drawing, *a* is a vessel furnished with a cover *b*, *c* revolving blades for mixing the materials, and *d* the cooling-jacket surrounding the vessel *a*.

It will be understood that I do not desire to confine myself to the use of the mixer herein shown, as any other suitable form of mixer may be used.

I am aware that insoluble nitrocellulose has heretofore been colloided in the presence of great cold in the standard solution of ether-alcohol—*i.e.*, two parts, by weight, of ether to one part, by weight, of alcohol—but so far as I am aware this substance has never before been colloided in ether alone.

As has before been stated, the nitrocellulose after being colloided has to be dried before it can be used as a smokeless powder. The word "dried" is used by powder-makers relatively, as experience has shown that it is impossible to get rid of all traces of the solvent, even after long periods of exposure to a high temperature. The ether-alcohol colloids are more difficult to dry than an ether colloid on account of the alcohol, which is itself less volatile than ether and which is permitted to and always contains as much as seven per cent. of water. A dried ether-alcohol colloid contains, residually, ether, alcohol, and water, and a dried ether colloid contains, residually, ether alone; but as the ether is more volatile than alcohol or water it is possible to dry an ether colloid more completely than an ether-alcohol colloid.

The nitrocellulose herein referred to as the lower limit of the insoluble nitrocelluloses, $C_{12}H_{15}O_5(NO_3)_5$, is the cellulose pentanitrate of Eder.

Having thus described my invention, I claim as new and desire to secure by Letters Patent—

1. An ether colloid of ether-alcohol-insoluble nitrocellulose possessing a content of nitrogen above that corresponding to the formula $C_{12}H_{15}O_5(NO_3)_5$.

2. The described method of forming a colloid consisting in subjecting insoluble nitrocellulose and ether to a temperature below that of freezing ethyl-alcohol ninety-five per cent. by volume absolute and in evaporating off the excess of solvent in the resultant product.

3. The described method of forming a colloid consisting in subjecting insoluble nitrocellulose and ether, in a closed vessel, to a temperature below that of freezing ethyl-alcohol ninety-five per cent. by volume absolute and in evaporating off the excess of solvent in the resultant product.

4. The described process of manufacture of a colloid powder consisting in subjecting insoluble nitrocellulose and ether in a closed vessel, to a temperature below that of freezing ethyl-alcohol ninety-five per cent. by volume absolute, mechanically agitating or kneading the cooled mixture, forming it into shapes or grains and drying.

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